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Gas Chromatographic Instrumentation

for

Gas Analysis of the Martian Atmosphere

FINAL REPORT

Volume III

Post Design Criteria and Summary

25 September 1962

Contract No. 950326

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JET PROPULSION LABORATORIES
Pasadena, California



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GAS CHROMATOGRAPHIC INSTRUMENTATION
for
GAS ANALYSIS OF THE MARTIAN ATMOSPHERE

FINAL REPORT

VOLUME III
POST DESIGN CRITERIA AND SUMMARY

25 September 1962

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SUBMITTED TO

Jet Propulsion Laboratories Pasadena, California

SUBMITTED BY

Melpar, Inc. 3000 Arlington Boulevard Falls Church, Virginia

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1. INTRODUCTION

This volume considers post design criteria related to the breadboard and prototype gas chromatography units. Attempts have been made to definitize all component parts of the package. A configuration of the gas chromatographic package with its contents is also presented. The package design takes into consideration environmental constraints.

Interspersed throughout this presentation are recommendations related to the injector valve, columns, detectors, sample pumping, and so on. Numerous trade-offs which could be made in increasing the versatility and, to some extent, the reliability of the instrument are also discussed.

Although this contract has been directed primarily towards the demonstration of the feasibility of the series column
configurations for doing the required analyses within the
Martian atmosphere, Melpar has directed a large portion of its
effort towards attempting to realize in a realistic manner
much of the hardware that would be needed in the breadboard
and prototype units. Many of the components evolved as a
result of this effort begin to approach the anticipated hardware. These items include such components as the detectors,
injection valve, atmospheric Venturi pump, and the gas
chromatographic oven with its chemical heaters.

2. OVERALL SYSTEM DESCRIPTION

In view of the excellent performance obtained from the laboratory unit, it is recommended that the breadboard unit have the same basic configuration. A schematic view of the proposed Martian gas chromatograph is shown in figure 1. proposed gas handling system is given in figure 2. The series column arrangement was used in the laboratory model, and is proposed for the breadboard model because of its theoretical These advantages were found to hold true in practice. A parallel configuration must use either a multiple injection valve which greatly increases size and weight, or it must use a stream splitter which is likely to be inaccurate and decrease sensitivity. The series system does not have these disadvantages. In addition, resolution of the last columns is improved by the partial separations made in the first columns. As an example, separation of Kr from ${\tt N}_2$ on a molecular sieve column is very difficult unless a silica gel column precedes it. An added advantage of the series arrangement is the proportionally smaller pressure drop across each column, which leads to improved separation efficiency. cross-section type detectors are used in the breadboard unit, because of the excellent reliability, sensitivity, and dynamic range found in the laboratory model. It may be seen that the basic system is almost identical to the laboratory model.

Column dimensions are significiantly changed. However,

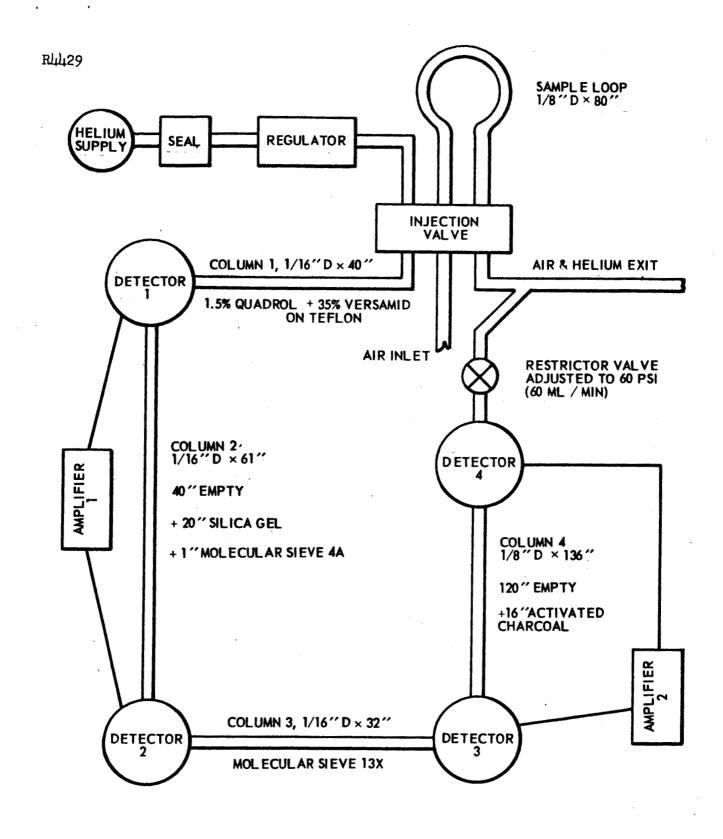


Figure 1. Schematic Diagram of the Breadboard Mars Gas Chromatograph

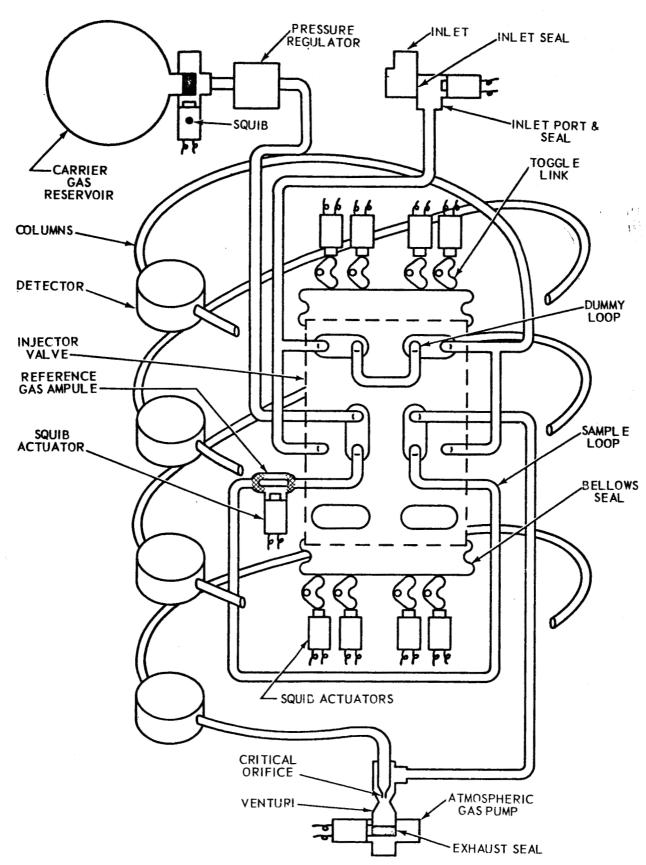


Figure 2. Flow Control System

the number of compounds analyzed and their order of elution will be the same. Improved resolution and sensitivity are expected, however. Some of the system components will be modified to correct minor difficulties experienced with the laboratory model. All of the components will be miniaturized.

- 3. DETAILED DESCRIPTION OF PROPOSED SYSTEM AND ITS COMPONENTS
- 3.1 Columns and Detectors

3.1.1 Columns

All columns will be constructed from smaller bore tubing than that used in the laboratory model. Smaller diameter columns are known to improve separation efficiency; thus shorter columns can be used. Smaller diameter columns naturally improve the velocity to volume carrier gas relationship. This improvement can be transmitted to the system as improved column efficiency, higher speed, or lower gas consumption. The breadboard unit has been designed to take advantage of this smaller diameter tubing, which has, associated with it, increased column efficiency and lower carrier gas consumption. The higher pressures to be used (120 psi inlet and 60 psi outlet) also will contribute to improved column efficiency.

Two slight difficulties may arise as a result of this modification. The decreased carrier gas volume will increase the injection time constant thus broadening slightly the elution peaks. Previous experience has shown that peak broadening is not excessive at 100 mb. pressure and 100 ml/min. carrier gas flow even when a 40 ml. sample loop was used. In any event, a sample loop smaller than the 15 ml. laboratory model could be used because detector sensitivity will be improved with lower flow rates. A 10 ml. sample loop will probably be more than adequate when a flow rate of 60 ml/min.

is used. Overloading of the columns will be more of a problem with smaller diameter columns. This is not expected to represent a limitation except for atmospheric components exceeding 10% concentration especially if a smaller sample loop is used. Two sample loops of different volumes may be conveniently used if desired.

All column tubing will be welded or brazed together, and to the detectors. This will eliminate the weight and potential leakage of fittings.

Column 1 is a liquid coated Teflon column designed to elute NH_3 shortly after air and $\mathrm{H}_2\mathrm{O}$ at five minutes. It is somewhat more alkaline than the laboratory model liquid phase column in order to prevent undesirable NH_3 adsorption previously experienced. It is as stable or more stable than the laboratory model. This is necessary to prevent column bleeding and resist the sterilization treatment.

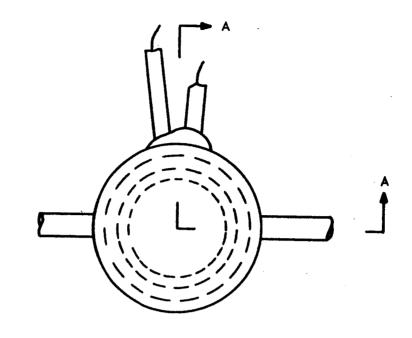
Column 2 is packed with a small amount of molecular sieve, a larger amount of silica gel, and is left empty at the front end. The empty portion serves to delay the passage of air into detector 2 until NH_3 has been eluted from column 1. Experiments are planned to attempt to change the separation of ethane and N_2 0. The molecular sieve portion of this column will be used to separate N_2 0 from CO_2 . This column will elute Kr, Xe, C_2H_6 , N_2O , and CO_2 in the 1.0 to 3.5 minute time period.

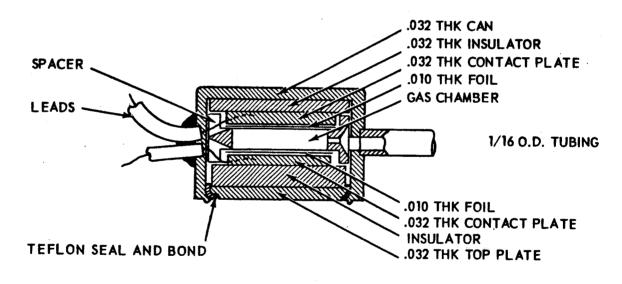
Column 3 is a conventional molecular sieve column. It is designed to elute H_2 , $A-O_2$, H_2 , Kr, CH_4 , CO, and Xe in the 1.1 to 4.5 minute time period.

Column 4 is partially filled with highly activated charcoal. This column is designed to adsorb O_2 and to elute H_2 , A, and N_2 in the 2.2 to 3.5 minute time period. The empty portion of the column serves to prevent interference with compounds being eluted from column 3. This column is constructed from larger diameter tubing in order to increase the oxygen adsorption efficiency and reduce the length of the empty portion of the column.

3.1.2 Detectors

Figure 3 illustrates the cross-section detectors that will be used in the breadboard unit. Melpar's cross-section detectors excel in response characteristics (see pp. 18-22 of the second monthly report). It is evident that the basic design is essentially unchanged from that used in the laboratory models. Columns are welded directly to these detectors rather than being connected with fittings and Teflon press fits. This will decrease weight and improve shock resistance. The electrode spacings will be graduated from 1/64 inch on detector 1 to 1/8 inch on detector 4 in order to compensate for the different pressures and gas velocities at each detector. This graduation will allow the use of a 10 volt power supply for each detector. The cell diameters will be decreased slightly to reduce space





SECTION A-A

Figure 3. Cross-Section Ionization Detector

requirements. More active tritium sources will be used to compensate for the reduced electrode area and to improve sensitivity. It is expected that these detectors will have the same high reliability that the laboratory models have, and will be considerably more shock resistant.

The detection sensitivity of the cross-section detector has been found to be adequate. With an effective tritium source of some 600 millicuries used in the laboratory model the detection sensitivities for all components with the exception of neon was near 10 ppm. The sample pressure used for these calibrations was 100 mb. There is no doubt that this sensitivity can be improved by a factor of 2.5 in insuring the proper response at 40 mb. A slight improvement in column efficiency, the use of a larger sample loop, and/or the use of a more potent tritium source would easily give the desired detection sensitivity at 40 mb. (The use of a more potent tritium source will not represent any exposure hazard. The energy of the tritium β -rays is small; and they do not penetrate the detector housing).

Estimated detection errors for the individual components at different concentration levels are given in table 1. These values were determined from the calibration curves in Volume II. The error was taken as five times the noise and drift at the particular current level measured. The limiting factor for accuracy at any one concentration level probably will be the

TABLE 1
ESTIMATED DETECTION ERROR

Concentration at 100 mb. pressure

Compound	0.001%	0.01%	0.1%	1.0%	10%
N ₂ O	±.0003	±.001	±.005	±.05	±0.5
с ₂ н ₆	.0007	.001	.005	.05	0.1
Xe	.0007	.0002	.001	.01	0.5
co ₂	.0009	.003	.008	.02	0.1
Kr		.0001	.001	.01	0.05
A	.0007	.0001	.001	.01	0.1
H ₂		.003	.001	.01	0,1
02			.005	.05	0.9
N ₂		.001	.005	.05	0.1
co	.0007	.003	.002	.01	1.8
CH ₄	.0003	.0002	.001	.02	

readout system rather than the detector. Overall accuracy for all compounds is affected by gas flow and temperature malfunctions. This error may be compensated for by inclusion of the standard reference compound.

3.1.3 Column and Detector Trade-Offs

A large number of major system changes in columns and detectors have been made during the development of the laboratory model. Some minor system changes and major mechanical changes have been made for the projected breadboard unit.

Other modifications would necessarily improve one characteristic at the expense of another. Most of these trade-offs for columns and detectors are listed in table 2.

A higher operating temperature (above 300°K) will reduce the retention time of all compounds, thus allowing longer and more efficient columns to be used. This would improve both separation and sensitivity because of the peak sharpening effect. It would, however, require more heater capacity and better insulation.

A larger sample loop would increase sensitivity to all compounds. The injection time would be correspondingly increased, and the peaks would be broadened slightly. Since no adverse peak broadening has been observed with the present sample loop when operated at 100 mb. pressure, a larger loop than the 15 cc loop considered may be used.

The present system could be easily modified so that no

TABLE 2
POSSIBLE MODIFICATIONS AND THEIR EFFECT ON THE SYSTEM

Modification	<u>Sensitivity</u>	Separation	Power & Wt.
Higher Temperature	+	+	-
Larger Sample Loop	+	-	0
Detector Switching	-	0	+
Higher Outlet Pressure	+		-
Higher Inlet Pressure	+	+	. -
Opposing Detector Polarities	+	0	-

Key: + = Beneficial Effect

0 = No Effect

- = Deleterious Effect

peaks, with the exception of Xenon, from amplifier 1 would interfere with those from amplifier 2. (See chromatograms in figures 5 and 6 of Volume II). This would make it possible to use only one amplifier if it is switched between detectors. A typical sequence of events would be: (1) Detectors 1 and 2 on, (2) Injection, (3) Air and NH₂ peaks from detector 1, (4) Detectors 1 and 2 off and 3 and 4 on, (5) H_2O_2-A , N_2 , Kr, CH_4 , H₂, CO, A, peaks from detectors 3 and 4, (6) Detectors 3 and 4 off and 1 and 2 on, (7) C_2H_5 , N_2O , CO_2 , H_2O peaks for detectors 1 and 2. The elimination of one amplifier by this method would represent a great saving in weight and space. The switching process would probably decrease stability, thus decreasing sensitivity to some extent. However, if this one amplifier fails all of the analysis would be lost instead of only half. A single amplifier may also be effectively used if the total analysis time is increased from five minutes to ten minutes, and column parameters are adjusted to spread the elution of components over the ten minute period. A ten minute analysis period would also enable H2S to be resolved by the liquid column.

Higher outlet pressures would improve column efficiency and thereby also improve sensitivity. But these higher pressures would require a higher gas flow to maintain the five minute analysis time.

Higher inlet pressures would lead to higher gas flows.

It would, however, enable longer and more efficient columns to be used which would lead to better resolution and detection sensitivity. The extra volume for the longer columns is objectionable to some extent particularly if the oven well must be enlarged to accommodate it.

Reversing the power supply polarities for the detectors so that they "buck-out" each other will greatly improve stability and sensitivity. Any adverse conditions that would tend to cause rapid drift would be nullified. This configuration will, however, require more complex electronic circuitry which will add to the weight of the package.

3.2 <u>Gas Chromatographic Oven</u>

3.2.1 <u>Introduction</u>

Successful functioning of a gas chromatograph is dependent upon an operational temperature, the same as that experienced near earth, of the order of 300°K or slightly higher. The payload of the Martian probe must be activated in a possible environmental temperature range of 140° to 300°K. Consequently, it is necessary to provide the chromatograph with a heating system capable of raising its temperature for an expected sampling period of 1 hour. The total power allocated to the chromatograph operation is 4 watts which is obviously not sufficient to accomplish thermal heating and regulation through electrical heating.

Due to the short duration of this contract effort, it was mandatory to develop the heating system for this mission simultaneously and independently of the effort devoted to the design of a laboratory model of the actual chromatograph instrumentation. Melpar elected to accomplish the heating of the unit utilizing the heat of reaction realized through chemical reaction.

The portion of the contract work necessarily used projected best engineering estimates of the final prototype unit as the basis for determining the weight, volume, and thermal properties of the components to be heated.

The design of any efficient or optimum heating system

is necessarily predicated upon an accurate knowledge of volume, mass, specific heat, and ambient temperature, none of which could be factually ascertained until after the finalized design has been resolved and fabricated. Melpar has completed a laboratory design of a heating system that definitely establishes the feasibility of designing a breadboard and production model gas chromatograph oven which meets the target specifications in every respect. It remains now to utilize the knowledge the data accumulated from this study to realize an operational breadboard unit during the next phase which will show optimum performance and which will integrate smoothly with the rest of the components of the gas chromatographic unit.

To fully exploit the potential of the chemical heating method developed during this effort, the design of the breadboard heating system can only be accomplished upon completion of the final breadboard chromatograph instrumentation. In this manner it will be possible to minimize both weight and volume and to provide for the most strategic possible location of the heating elements relative to the individual components of the apparatus.

Thus, a larger quantity of smaller heaters would permit both better thermal control and more space for either electronic components or insulation. Furthermore, such a change would also result in a reduction in overall weight

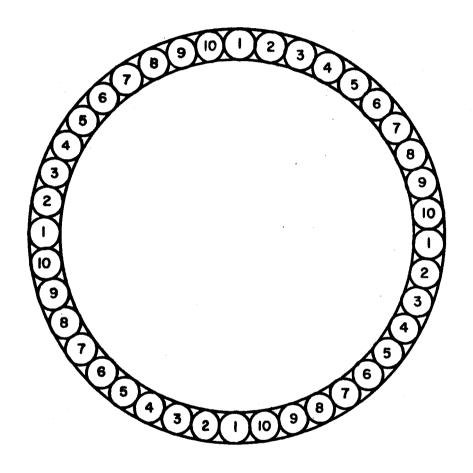


Figure 4. Firing of Smaller Chemical Heaters in Sequence Four at a Time

of the actual heating system. Presently, the heating system composed of heating elements and aluminum cylinders as shown in figure 1 of Volume II weighs approximately 1.5 pounds. The proposed change would reduce this to less than 1 pound. An improved, controlled, chemical reaction for heating the oven could reduce slightly even this weight.

It would be extremely difficult and rather impractical to attempt to present a firm heating system design for the breadboard unit at this time. Design changes with the oven, however, are quite flexible; the oven may be readily fabricated to accommodate the components it must contain. Design considerations that are involved in the breadboard oven are disussed in the following paragraphs.

3.2.2 Chemical Heaters

Close examination of the laboratory model of the heating system immediately reveals one rather undesirable feature; the heat is released from each charge at only one point on the circumference of the chamber at one time, resulting in a rather severe gradient throughout the chamber and at the same time increasing equilibrium time. This situation can be improved and the overall thermal gradients improved by reducing the size of the heaters and increasing their number in realizing the same amount of heat. A typical example of the multiheater arrangement is shown in figure 4. In this case the heat would be produced by 40 chemical heaters

rather than 12, and 10 groups of 4 would be fired in sequence instead of as individual heaters.

A reduction in heater diameter such as this would also allow a reduction in the overall diameter of the heater chamber. Presently, the outside diameter of the chamber is 4.375 inches to accommodate the 3-inch I.D. well required for the injector valve, columns and detectors as well as the chemical heaters. A reduction in the diameter of the chemical charge from the present 0.375 inches to 0.176 inches would permit the outside diameter of the chamber to be reduced to 3.6 inches. In the event that the columns could be contained, along with the valve and detector in a still smaller chamber, say 2.5 inches in diameter, the OD of the chamber could be reduced still further to 3.1 inches.

Another factor that warrants consideration in the overall efficiency of the heating system is the actual lowest outside ambient temperature to be experienced in flight. The specification for this unit requires that it be capable of surviving a low temperature of 173°K. At the same time, it also specifies that the sampling shall commence in an atmosphere at 140°K. If the lower temperature limit were indeed 173°K, a considerable saving in the amount of heat (and hence the heat packet required to establish thermal equilibrium at the operational temperature) could be realized. For a minimum temperature of 173°K, it would be possible

to reduce the amount of chemical change by 20% thereby reducing the weight of the heating system by 20%. More important, however, such a change might also allow a reduction in the amount of insulation required and a consequent increase in the amount of space available for additional components.

The feasibility study has proved that an oven temperature near 300°K can be realized, even if a 140°K environment does exist; but Melpar does recommend a further study of transit temperature in order to realize the most efficient breadboard unit possible.

3.2.3 Thermal Insulation

The heat leak in the laboratory model of the heating system is not excessive, for it is possible to achieve the desired temperatures and maintain them for a considerably long period of time. In the final application, however, it may be necessary to find a means of obtaining better insulation for the unit when it is integrated with all components of the gas chromatography instrument. During the preliminary screening of such materials for use in this system, Melpar did not attempt to use the so-called superinsulators. Foamed materials could more easily be used in the laboratory during this phase of the work than could the superinsulators. As a result of a literature search and a conference with personnel at NBS, Boulder, Colorado, two factors that definitely affect the use of superinsulators

became apparent: (1) such material has not been used in thicknesses of less than ½ inch, and (2) there is no data on the performance or actual conductivity above liquid nitrogen temperatures. Although the radiation transfer is extremely important at 77° K, it may well be that these insulators are rather poor in the region from 200° to 300° K. Still another factor that must be considered is the problem of the vacuum required, and the construction necessary to hold low pressures for several months. Figure 5 shows a graph of a number of these insulations as a function of vacuum. For comparison purposes, the insulfoam presently used has a thermal conductivity of approximately 210 μ watts cm/cm² $^{\circ}$ K. To design the prototype as efficiently as possible, Melpar will consider these insulations further in the next phase.

One method for improving the insulation properties of the foamed insulations also remains to be considered. This is the effect of vacuum upon the foamed insulations. Since the laboratory model has been packaged in a hermetically sealed can, it would also be possible to evacuate the whole unit in order to lower the conductivity. This may prove to be a good compromise between the superinsulators and the present foam.

3.2.4 The Integrated Oven Package

The packaging of all of the laboratory components --

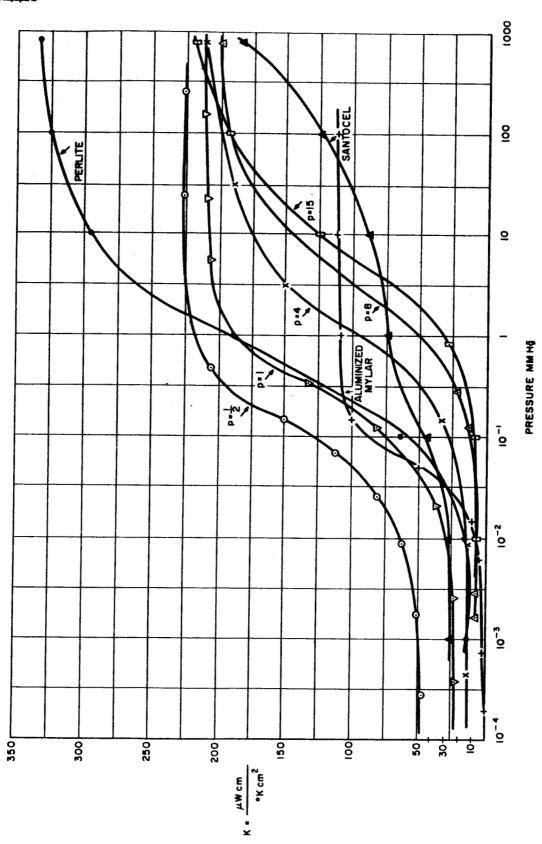


Figure 5. Thermal Conductivity of Several Syper Insulators as a Function of Vacuum or Pressure

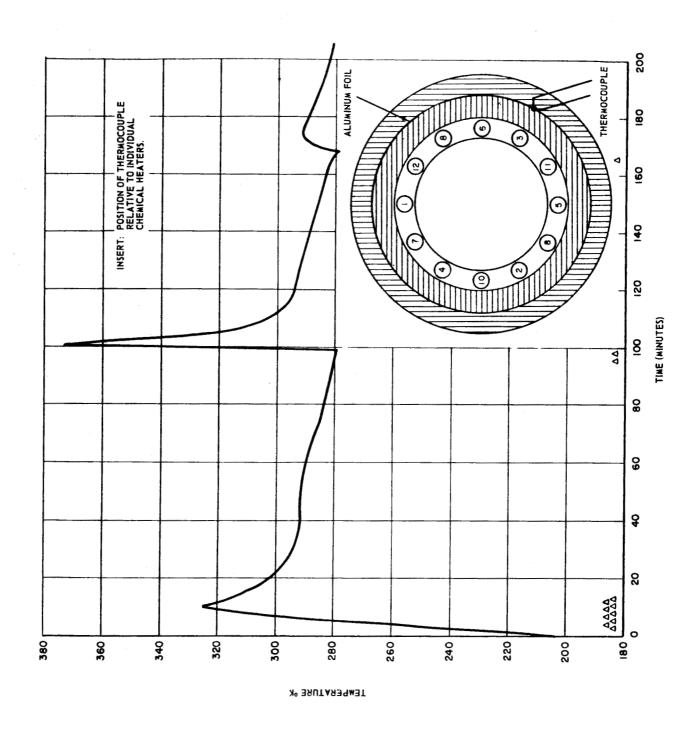


Figure 6. Temperature at a Point Between Layers of Insulation

chromatograph, electronics, and heating system--into one integrated unit will require the efficient utilization of every cubic inch of space.

Since the solid-state electronics to be used in this apparatus require temperatures of the order of 250°K for operation, Melpar will make use of the heat escaping from the chromatograph chamber to provide the necessary environment.

Figure 6 shows a graph of the temperature between the two 3/8-inch-thick layers of insulation used in one of the chemical heating experiments. The delta values represent the times at which the chemical heaters were fired. The insert sketch shows this sequence and the location of the thermocouple in this insulation. The modification of the heating system, as proposed, to fire four small charges in place of one big charge would even out this curve and reduce the peak temperatures to a much more tolerable situation. On this basis, it would seem quite feasible to mount the components on curved printed circuit boards at this point and in the corners.

Figure 7 shows a sketch of the areas that could be utilized for this purpose. As shown, some 62 cubic inches are available. A more detailed consideration of the overall packaging will be given in a later section.

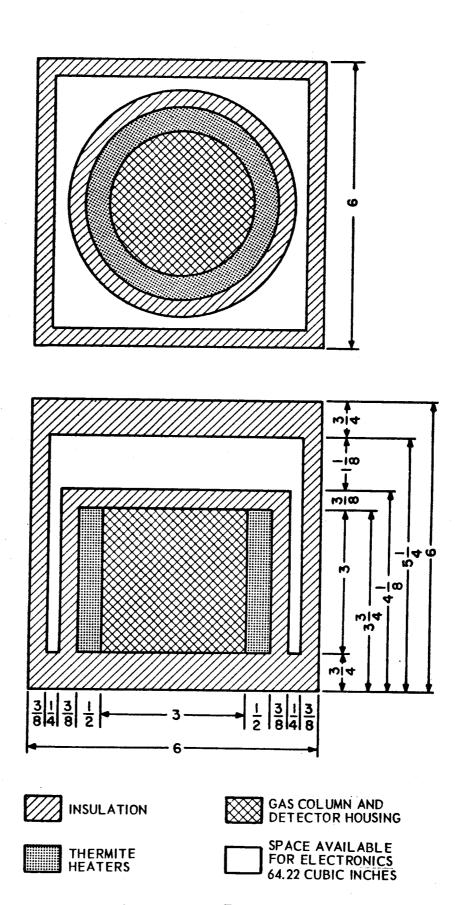


Figure 7. Oven and Its Enclosure

3.3 <u>Mechanical Components</u>

3.3.1 Introduction

The satisfactory design and development of a bread-board gas chromatograph for the Martian atmosphere will depend heavily on a satisfactory design for a carrier-gas and atmospheric gas-flow-control system. It will be necessary to store a carrier gas for the chromatograph and to supply the Martian atmosphere gas and provide satisfactory means of injecting the sample gas into the columns and detectors. The design of this system will be basically divided into general categories including the carrier gascontrol system, the atmosphere gas-control system, the injector and columns, and the squib operation control. Each of these areas involve particular design problems, and the satisfactory interrelation and integration of the systems will be necessary to provide a reliable flow-control system.

3.3.2 General Considerations for the Flow System

To establish the design characteristics of components in the system it is necessary to assume certain parameters of gas flow and pressure for operation of the chromatograph. These parameters have been established based on an extrapolation of the experimental work with the laboratory model. At the critical orifice, which is placed at the end of the system, a flow of 60 millileters per minute will be required with a pressure drop of 60 psia. Ambient atmosphere pressure

changes will not affect this pressure because of the characteristics of the critical orifice. The columns will require a head pressure of 120 psia, and assuming isothermal expansion, this will reflect a flow at the entrance to the columns of 30 millileters per minute. For purposes of preliminary calculations the pressure drop through the injector valve is assumed to be negligible.

A severe temperature change is expected to be experienced between the injector valve and the pressure regulator because the injector valve will be enclosed within the thermite oven and the pressure regulator will be external to the oven and its insulation. Assuming adiabatic expansion with a temperature of 300°K at the valve, and a temperature of 140°K at the regulator, the required flow rate at the regulator will be 14 milliliters per minute. The necessary pressure is assumed to be 120 psia at the regulator and nearly constant to the head of the columns. The pressurized carrier gas tank is also assumed to be at the same temperature as the regulator, that is 140°K. If the tank is filled to a pressure of 6000 psia at room temperature (300°K), the pressure at the low temperature would be approximately 2800 psia. Again assuming isothermal expansion, the flow leaving the tank would be 0.6 millileters per minute. The last assumption to be made is that the flow of 0.6 millileters per minute from the tank will be required for a period totaling 80 minutes.

event that the temperature of the regulator and tank are higher than the expected 140°K, the flow rates will be higher at these points but will be compensated by the adiabatic expansion into the injector valve.

3.3.3 Carrier Gas-Control System

The carrier gas-control system will consist essentially of the pressure tank and a squib-operated seal to open the tank upon entry into the Martian atmosphere, and a pressure regulator to provide the helium flow at the proper pressure to the injection valve and the columns. During the course of the research phase on this program the availability of pressure tanks as well as miniature regulators was investigated. It was determined that several sources for the procurement of the pressure tank fabricated in titanium metal were available as well as several sources for miniature regulators.

3.3.3.1 Pressure Tank and Seal: The useable volume of the pressure tank which would supply 0.6 milliliter per minute for helium for a period of 80 minutes would be approximately 48 milliliters, or near 3 cubic inches. Considering this component individually, the optimum configuration for a maximum supply of helium with a minimum weight of the container tank would be a sphere. The use of titanium metal for the construction of this sphere will also provide the best weight-strength ratio practical. This is the configuration which is

proposed, and the internal diameter of this sphere will be 1.79 inches. Because the tank as well as the remainder of the chromatograph system will be subjected to sterilization temperatures of 145°C, it will be necessary to assure that the tank will withstand the increased pressure due to the increased temperature. Preliminary calculations indicate that this pressure may be as high as 1800 p.s.i.a. Therefore, the minimum required thickness of the tank wall will be 0.065 inch.

Figure 8 shows a design concept for a pressure tank with an integrated squib actuator seal device. The tank will be constructed in two hemispheres. One hemisphere will have a protruding cylindrical tip with a closed end which will be placed in a position to be ruptured by the squib actuator. The second hemisphere will contain the filling port which will be sealed after the helium filling is completed. The two hemispheres will be welded together and ground smooth to ensure that no high stress points develop because of sharp edges or indentures. Figure 8 shows the unit both before firing the squib and after firing the squib. The squib will be constructed in such a manner that a bellows seal between piston and cylinder will prevent any of the gases resulting from the squib action from entering the helium stream. exit port of the pressure tank and seal will be connected directly to the pressure regulator. Sources contacted by

AFTER FIRING

APPROX. FULL SIZE

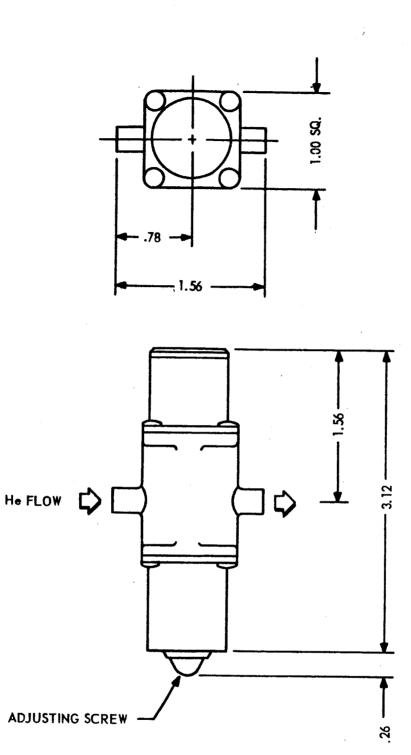
Figure 8. Pressure Tank and Seal

Melpar in connection with supplying the titanium pressure vessel are as follows: the Menasco Manufacturing Company, Burbank, California, and the Brooks and Perkins Company, Detroit, Michigan.

The tank may be filled by cooling it at a low temperature (such as liquid nitrogen) and then filling it at available tank pressures. If the charging pressure is proper, a pressure of 60 psi will be realized when the sphere is heated to room temperature.

3.3.3.2 Requiator: Most common regulators are designed to utilize a large diameter flat diaphram operating in conjunction with a needle valve and pressure spring to allow a buildup to a predetermined pressure before closing. Miniature valves utilizing this design are available which would meet the pressure regulation and temperature requirements for this project. However, the basic configuration does not particularly lend itself to a suitable arrangement with the overall gas chromatograph unit. One supplier of this type of valve is the Sterer Engineering and Manufacturing Company, North Hollywood, California.

Figure 9 shows a design utilizing a slightly different principle of operation which will provide a suitable configuration and at the same time meet the requirements of pressure regulation, temperature, and miniaturization required for this program. This device is available from Wallace O.



SUGGESTED SOURCE WALLACE O. LEONARD, INC. PASADENA, CALIFORNIA

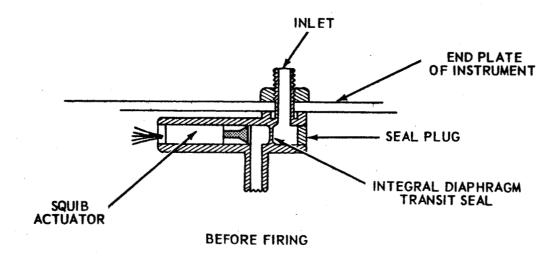
Figure 9. Pressure Regulator

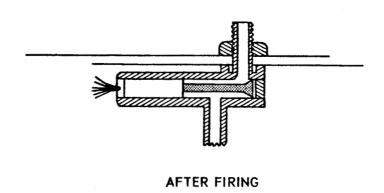
Leonard Inc., Pasadena, California. Both of these companies have been contacted by Melpar to ensure the availability of the devices. It appears at this time that the configuration used by the Leonard Company would be more suitable; their valve is therefore considered to be the type to be used in the breadboard unit.

3.3.4 Atmosphere Gas-Control System

Two general problem areas in the atmosphere gascontrol system involved the sealing of the system from external
contamination prior to injection at the Martian atmosphere
and a method of ensuring passage of the atmospheric sample
through the sample loop after the vehicle has hit the surface
of Mars. It is proposed that the flow-control system be
flushed and filled with helium prior to final assembly. At
this time a positive hermetic inlet seal will be provided
as well as a positive hermetic exhaust seal. Both of these
seals will be ruptured by squib actuators when the instrument
is activated.

3.3.4.1 <u>Inlet Seal and Manifold</u>: Figure 10 shows the concept envisioned for the prototype design for the inlet seal. An offset-type seal is used here merely to give more flexibility in the placement of the device in the end panel of the instrument. A short length of tubing constituting a manifold will lead from this inlet port seal to the injector valve in the oven.



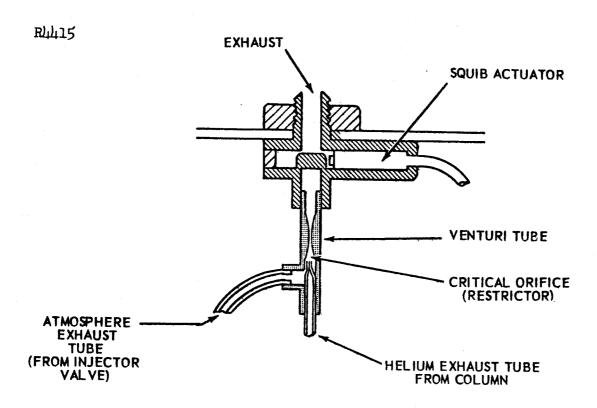


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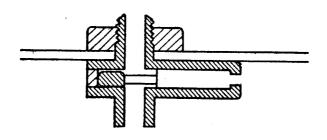
Figure 10. Inlet Port and Seal

If the vehicle uses ablative materials for atmospheric entry, it would be desirable to ensure that total utilization of the ablation materials occurs prior to instrument sampling. If a scoop is used for sample pickup it should be sealed closed until ablation is complete and then released by explosive bolts or squib latches. The manifold leading from the scoop to the instrument inlet port should be short and nonrestrictive. In addition, the scoop inlet and exhaust port in the capsule must be so located and designed to ensure passage of the atmospheric gases after impact.

The unit will be designed to provide one integral exhaust for both the atmospheric gas sample loop and the effluent from the chromatographic columns. With such an arrangement a Venturi tube in the exhaust will effectively pump atmospheric sample through the sample loop after impact. A critical orifice will be incorporated into the Venturi in controlling the carrier gas flow through the columns. It is anticipated that this device can be miniaturized as shown in figure 11 (full size). The atmospheric gas will be directed through a manifold from the sample loop outlet in the injector valve to a side port in this gas pump; and the column effluent will be fed through the critical orifice and then directed at the throat of the Venturi tube. Referring to the report on the laboratory model (Volume II) it may be seen that with the higher pressure at the critical orifice, 60 psia, a considerably



BEFORE FIRING



AFTER FIRING

APPROX. FULL SIZE

Figure 11. Exhaust Seal and Gas Pump

larger flow of the atmospheric gas may be expected through the sample loop. Although the 4-to-1 flow ratio (sample to helium flow) may not still hold true, the flow will certainly be sufficient. Downstream from the gas pump a squibactuated seal will be placed directly in line with the axis of the pump. This will be an hermetic seal and will be squib actuated as shown in the figure 11. It is desirable to arrange the placement of the gas chromatograph instrument in the capsule in a manner so that the exit flow from this gas pump to the outside atmosphere will not be particularly restricted or inhibited. Some small restriction due to the length of manifold or a few turns may be acceptable.

3.3.5 Sample Injection and Columns

To minimize temperature change effects on the chromatographic analysis by the injector valve, the injector valve will be placed in the oven with its axis parallel to the axis of the coil of columns and sample loop.

3.3.5.1 <u>Injector Operation and Sealing</u>: The injector valve which is proposed for use in the breadboard and prototype models is identical in operation with the unit used on the laboratory model with the exception that the slotted surface which was used in the Teflon slider is now a spool section which will not require a backup spring. Figure 12 shows an exploded view of the injector valve proposed. All of the ports leading into the valve will come in through the

baseplate and enter the cylindrical section in the center of the valve. This cylindrical section will be lined with a Teflondeposited surface. This Teflon surface will be polished to provide a fine finish sealing surface with the stainless steel spool. The stainless steel spool will contain the slots for interconnection of the ports upon actuation.

These stainless steel spools will be finished as well to a fine mating surface with the Teflon deposited lining. The combination will provide long leak paths in any direction as well as minimize the problems of Teflon cold flow. After the valve spool has been inserted and locked in position with a locking key to prevent rotation, metallic bellows will be placed on each end of the block to provide hermetic sealing of the valve actuation section from the external area.

Figure 13 shows a cross section of the proposed design for this valve. It may be seen that the size of the valve has been reduced considerably from the laboratory model and that the unit is now constructed of few pieces. As a result of experimental work with the laboratory model it was found that a pressure change effecting the operation of the columns and detectors resulted when switching from the sampling position to the inject position because of the pressure drop through the sample loop. Therefore, a dummy loop has been added to the valve which will provide a compensating pressure drop in each position of the valve. This arrangement also

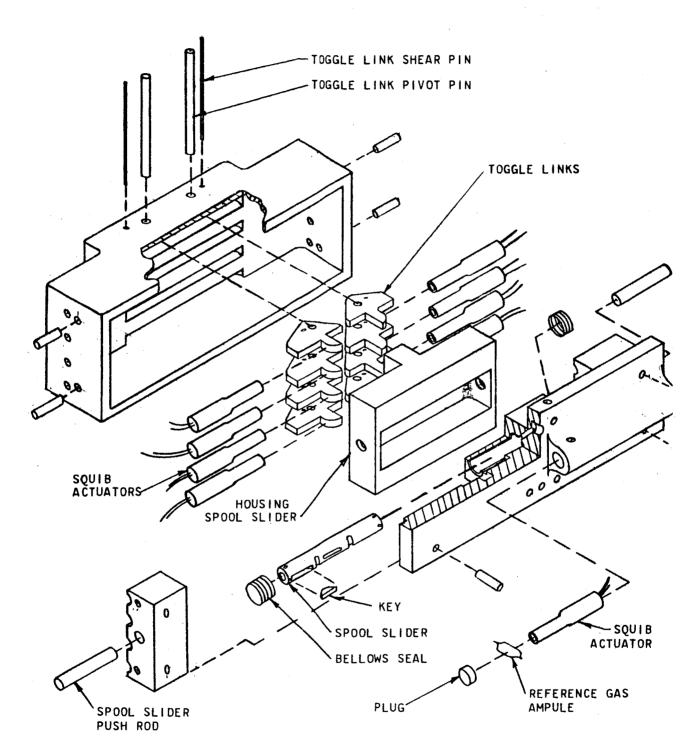


Figure 12. Sample Injection Valve

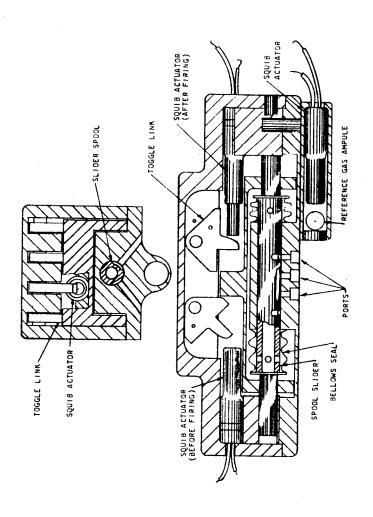


Figure 13. Cross Section of Injection Valve

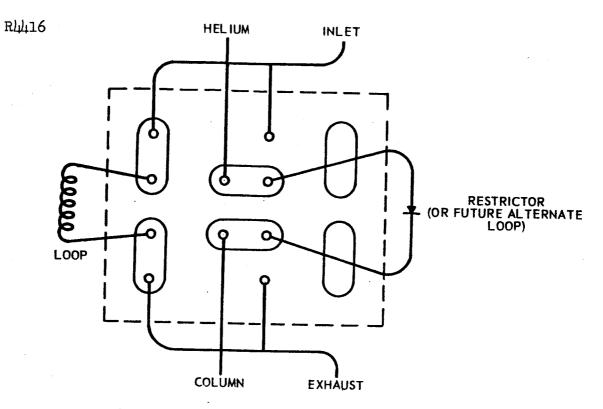
provides for the addition of a second sample loop if this should at some future point prove desirable. The arrangement of these ports and interconnecting slots in the spool are shown in figure 14.

3.3.5.2 Column Interconnection and Sealing: The proposed design provides for the wrapping of both the columns and sample loop in a concentric form within the internal diameter of the oven. With the injector valve placed off center within this helix the detectors will be interconnected between the top of the injector valve and the opposite side of the helix columns and sample loops. A slightly sharper radius in the tubing coming off the helix will provide interconnection to the detectors. It is intended that all joints in the system be hermetically sealed. One method of accomplishing this which will be the use of ceramic- or alumina-deposited tube ends and joining of these ends by means of brazing. Tubing connections which are required to go through the oven walls will have transition sections of insulating material bonded to the tube ends.

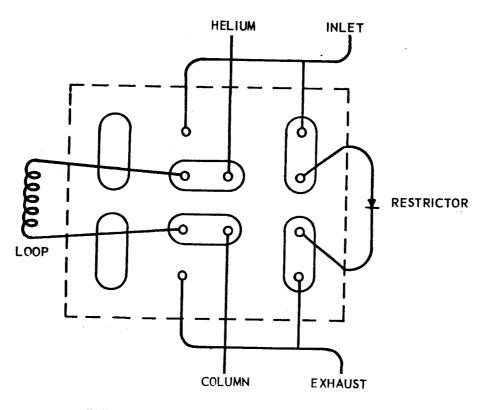
The columns therefore will be sealed within the limits of the flow system. That is, entrance and exhaust, carrier gas tank, pressure regulator, injection valve and all tubing will be sealed for transit.

3.3.6 Squib Operation Control

Extensive utilization of gas squib actuators in the



INJECTOR VALVE IN SAMPLING POSITION



INJECTOR VALVE IN INJECT POSITION

Figure 14. Valve Port Connections

breadboard and prototype designs provides a high degree of efficiency of available power utilization. For example, the Atlas Chemical Co. squibs which are approximately 1-inch long and 1/2-inch diameter, will produce a 5/16-inch piston stroke with a force of 60 lbs. This is produced with an input voltage of 18 volts across 10-ohms resistance for a matter of 3 to 5 milliseconds. To preclude pulses due to the sudden power drain of these squibs, an RC circuit is proposed to be used directly with the 28 vdc power in the capsule. This circuit as well as the squib interconnection is shown in figure 15. After initial charge, the circuit would not draw current except at a slow rate in recharging the capacitor after squib firing.

Referring to figure 15, note than an initiator squib switch ensures that all squib leads are short circuited prior to functioning. A fuzite-type (fuze wire spring) switch is used to short the initiator squib. Upon receiving a command start pulse the fuzite switch causes the initiator squib to fire thereby breaking the short on all other squibs and making proper connections to the master programmer. Squibs A_1 and A_2 may be fired simultaneously or in close sequence to admit the sample gas. Squib B_1 is fired next to initiate the carrier gas flow. Squib B_2 for the reference gas sample may be fired with the first, or any other sample injection desirable. Squibs C_1 , C_3 , C_5 and C_7 are fired at the beginning of each

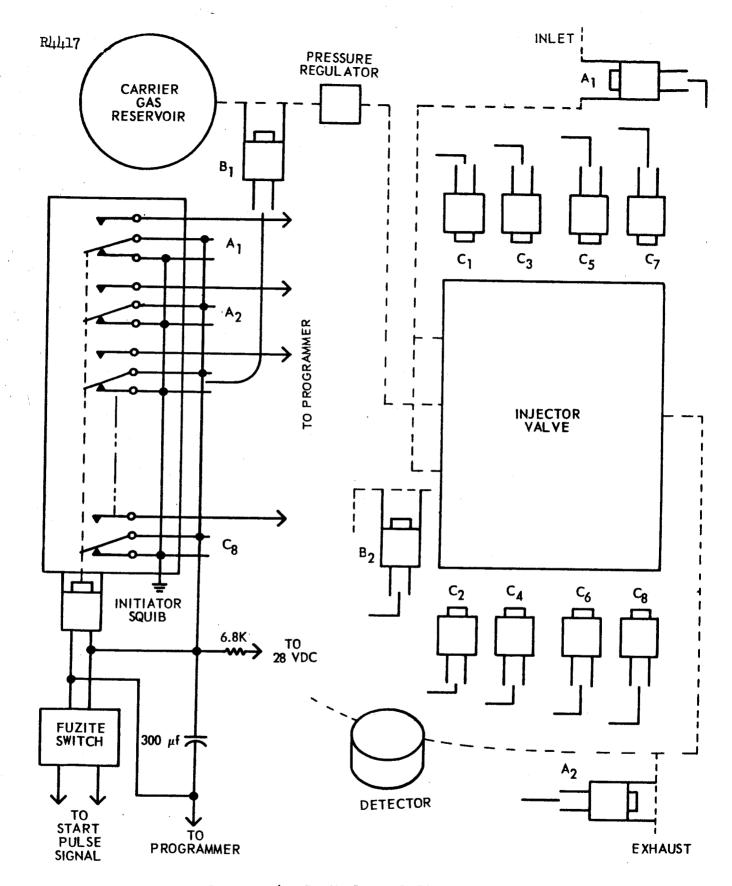


Figure 15. Squib Control Circuit

sample injection to direct the carrier gas through the sample loop. Squibs C_2 , C_4 , C_6 and C_8 are fired to accomplish the return strokes of the injector valve.

3.4 <u>Electronics Post Design Considerations</u>

3.4.1 <u>Introduction</u>

This initial study for a Mars Gas Chromatograph in the electronics field has resulted in delineation of the concept, and practical demonstration of a completely solid-state electrometer package, which is capable of operating in an optimum manner on the outputs of the detector cells to produce a 0 to 5 volt signal proportional to either the detector cell's current or the logarithiem of this current. Likewise, all of the ancillary circuits associated with the design, viz, power supplies and regulators, logarithmic amplifiers and programmer are selected to utilize 100% solid-state components. The power drain of the individual electronics components that are now estimated for the breadboard is shown in table 3. (Note that the 1.5 watt power requirement leaves 2.5 watts that may be used in oven heating if desired).

The problem now arises as to the applicability of these designs to the weight, power and reliability requirements of the equipment that must actually be launched into space, ability to survive the rigors of the launch, the voyage through space, entry into the Martian atmosphere and impact (even though it is easy) on the surface of the planet. The basic choice of solid state design is considered to be a good start for minimizing weight and power consumption and insuring

TABLE 3
POWER CONSUMPTION FOR ELECTRONICS

2	Electrometer Amplifiers		300 m	illiwatts	
2	Logarithmic Amplifiers		170	11	
2	Feedback Amplifiers		170	π	
1	Detector Cell Power Supplies		20	11	
1	Electronics Power Supply		600	††	
1	Programmer I		50	Ħ	
1	Programmer II		150	Ħ	
		Total	1.46	watts	_

a design capable of superior reliability in the face of severe shock and vibration. Every effort has been taken to minimize the use of rotating and translational mechanical components to further move toward the goal of the ultimate in mechanical reliability.

For the next step of this program, it is recommended that the basic designs which have been established be carefully reviewed, and where necessary appropriately modified to realize fully the potential of the inherent mechanical and electrical characteristics of the concepts involved for production of a system of minimum size, weight, and power consumption and possessing high reliability. It is recommended that these designs be breadboarded and tested to experimentally ascertain their capability to perform satisfactorily after being subjected to the rigors of the space voyage, both during the transit through the Martian atmosphere and after soft impact on the planet's surface.

In the following sections, consideration of the general environmental conditions are presented and the design philosophy and possible alternate approaches are considered for the electrometer amplifier (including feedback amplifier), logarithmic amplifier, power supplies, programmer, and squib control circuit.

Finally, a discussion concerning the data processing is presented. This subject is reviewed in light of the findings

made during this study.

3.4.2 <u>General Environmental Considerations</u>

Before proceeding with the discussion of specific components of the electronics subsystems consider the general nature of the environment to which the system must be subjected. The components must be capable of surviving without any deterioration in the heating and gaseous atmosphere of the sterilization. It is anticipated that during one phase of this treatment the temperature will be maintained at 145°C for 36 hours and this treatment will be repeated a number of times. The equipment will then be subjected to a biological sterilization gas, probably entylene oxide and hence, for a period of approximately 11 hours at 20°C and 35% relative humidity. It is also possible that certain liquid sterilants may be employed. For these sterilization treatments, the electronics must be designed to survive in the non-operative state without any deterioration.

From the point of view of shock and vibration the equipment must be capable of surviving the shocks expected during handling, the vibration expected during transportation, the static acceleration and vibration of launch, the static deceleration of entry into the Martian atmosphere and the shock of a soft landing on the Martian surface. It is expected that static accelerations of +14 G and -6 G may occur during launch and Martian atmosphere entry, that random vibration of

15 G for 6 seconds, 10G for 180 sec and 4½ G for 360 seconds, using a white gaussian noise source with a bandpass from 15 cps to 1500 cps and that shocks of 200 G with peak durations of 0.5 to 1.5 milliseconds must be endured without damage.

Electrically, the equipment should be expected to survive unharmed OVER-VOLTAGES of possibly as great as 70% and to exhibit R-F and magnetic field interference levels no greater than those currently stated in the specifications for the subject contract, viz, the magnetic field must not exceed 1 gamma at 3.5 feet from the gas chromatograph package and no R-F radiation capable of interferring with other units of the package shall be emitted. To meet these requirements, precautions in wiring involving the use of recognized constriction disciplines, such as twisted pairs, proper bonding, shielding, etc., to minimize interference due to conduction (this is particularly important in the high impedance portions of the detector-electrometer system), electrostatic induction, electromagnetic induction, cross-talk, junction of thermal potentials, and ground loops will be strictly adhered to. One point of crucial importance is the potential which can be generated by mechanical vibration of the high impedance leads interconnecting the detectors and electrometer amplifier. Vibration of these leads can produce significant undesired perturbation in the electrometer output. To avoid this, the connections involved must be kept as short as possible and be constricted in a very rigid manner. Flexible leads are probably not satisfactory to this application.

Concerning temperature, it is expected that during nonoperating conditions the temperature may range from a high
of 73°C to a low of -133°C. Components must be selected to
survive temperatures within these limits in non-operative
conditions without damage with the possible exception of
the programmer. Since the programmer must initiate the system
WARM up, it may have to OPERATE at a temperature close to the
minimum stated above. Temperature conditions for operation
of all other electronic components may be somewhat relaxed
since advantage can be taken of the fact that the oven must
warm-up and some of the energy will probably be available for
warming up the electronics units.

3.4.3 <u>Programming</u>

It will be necessary to control the succession of operations performed in the gas chromatograph sonde. These operations fall in two relatively distinct classes each logically requiring separate programming functions. Hence, for the purpose of this discussion one may think of the overall programmer as being comprised of two programming units. These have been designated as Programmer I and Programmer II, respectively, in the discussion which follows.

The function of Programmer I is to initiate and control the system warm-up. The function of Programmer II is to

initiate and control the events pertinent to the actual chromatographic analysis. Typical of these functions are electrometer amplifier initial zeroing and activation of the sample injection valve. Operations performed by the two programmers are detailed in table 4. Design of the circuits for all solid-state programmers are discussed in the following paragraphs.

3.4.3.1 Programmer No. I: For heating the package, at present, twelve chemical heaters are provided. Each has good thermal contact with the case and will distribute its heat to the package rapidly. Nevertheless, a finite time interval must elapse after firing each heater before firing the next to insure distribution of heat to the thermostat. A tentative interval of ninety seconds was derived experimentally. The present chemical igniters each require 500 ma at three volts for ten seconds to insure ignition. The intense momentary heat may cause shorting of the electrodes within the chemical cartridge. To prevent this from resulting in an undue load on the battery the ignition current should be interrupted after ten seconds.

Programmer No. I will consist of a miniature thermostat that senses the temperature of the injection valve. As long as this thermostat indicates temperature below operating range, it will supply signal to a pulse generator. Upon receipt of this signal and each ninety seconds thereafter, while the

TABLE 4

PROGRAM SCHEDULE

<u>Time</u>	<u>Function</u>
0 to 20 minutes	Fire thermitic units
20 minutes	Break seals on: 1. helium supply 2. column exit 3. sample entrance
25 minutes	Energize power supply and amplifiers
39 minutes	Zero baseline
40 minutes	Inject first sample
40 to 45 minutes	Analyze first sample
45 minutes	Return sample valve for 2nd injection
45 to 50 minutes	Flush columns and acclimatize sample loop
50 minutes	Zero baseline
51 minutes	Inject second sample
51 to 56 minutes	Analyze second sample
56 minutes	Return sample valve for 3rd injection
56 to 61 minutes	Flush columns and acclimatize sample loop
61 minutes	Zero baseline
62 minutes	Inject third sample
62 to 67 minutes	Analyze third sample
67 minutes	Turn off

signal remains, the pulse generator will produce an output pulse. The pulse will be fed into a one-shot pulse stretcher which will extend its duration to ten seconds and pass it to an electronic commutator or multiplexer, one output of which will be to each igniter. Each pulse entering will ignite only one chemical heater. Upon secession of each pulse the multiplexer will step to the next state so that the following pulse will ignite the next heater. A block diagram of Programmer No. I is shown in figure 16.

The Programmer I circuit involves the use of transistor flip-flops and gates sequenced by a pulse advanced along an electronic solid-state shift register. All of the circuits involved are of the binary type and can be fabricated using commercially available silicon transistors to meet the environmental conditions anticipated.

A possible alternate approach might incorporate a pulse generator operating an electromechanical stepping switch. However, it is probable that such a design would be of greater overall weight, possess no power consumption advantage and be less reliable.

3.4.3.2 <u>Programmer No. II</u>: The timed functions are mostly of the type requiring initiation only. The duration of the start pulse is unimportant. The firing of squibs to actuate the injection valve are timed functions of this nature. Other operations require controlled finite duration



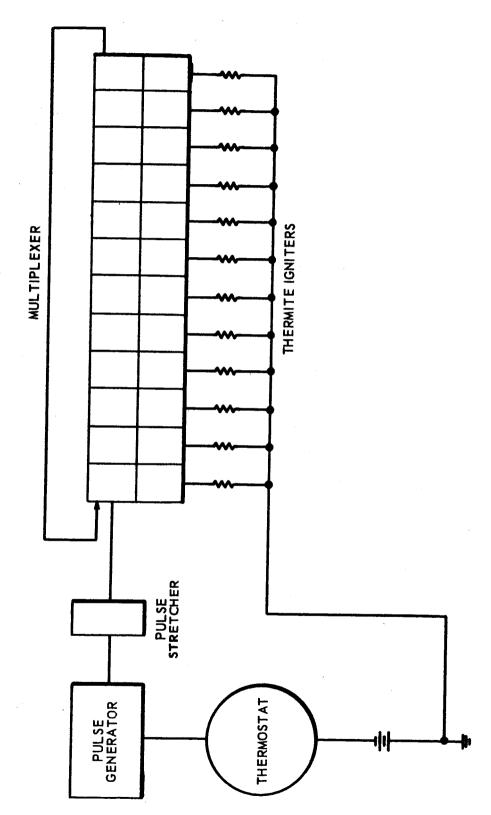


Figure 16. Block Diagram of Programmer I

such as the zeroing of the electrometer amplifier baseline. To control these functions a binary electronic clock has been The electronic clock will consist of a free-running multivibrator operating at about eight cycles per second. This multivibrator will be frequency stable to about one part in one thousand or better and will be the primary time reference for the gas analysis system. The multivibrator will drive the first of a string of fifteen binary frequency divider flip-flops. In order that changing load conditions will not affect the frequency stability of the multivibrator, the first flip-flop will be its only direct load. puts of Programmer No. II are derived from coincidence (and) gates, the inputs of which are the flip-flop outputs. case of momentary outputs, the gate itself or a driver amplifier following the gate will suffice to drive the load. case of a sustained output, two coincidence gates will be used. One will set a flip-flop to initiate the operation; the other will reset the same flip-flop to terminate the operation. Where the same operation is to be performed more than once. "or" gates will permit setting and resetting of flip-flops more than once during the gas analysis procedure. A block diagram of Programmer No. II is presented as figure 17.

For example it might be required to initiate some function at 24 minutes and terminate at 24 minutes and terminate at one minute later. That is: it starts at time 0 + 1440 seconds and

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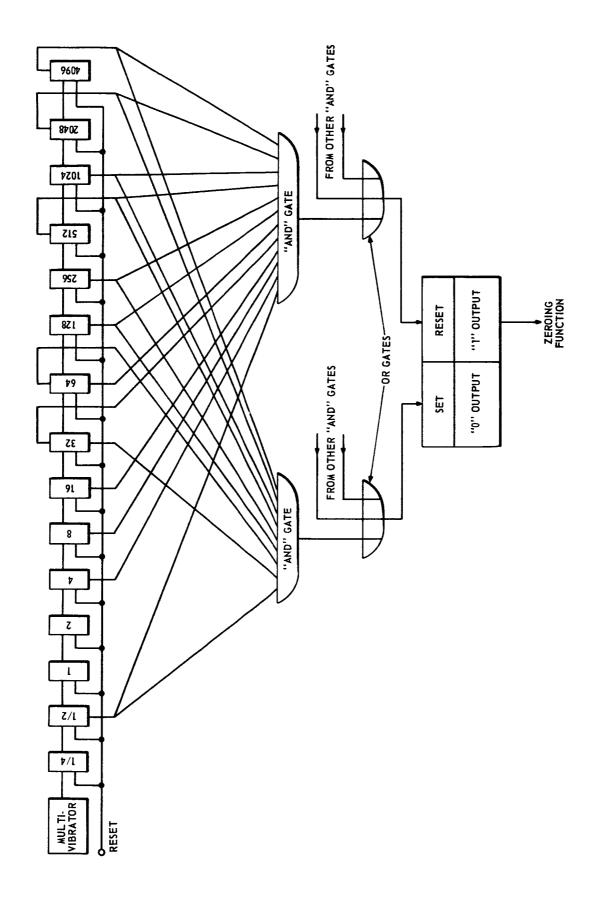


Figure 17. Block Diagram of Programmer II

terminates at 0 + 1500 seconds. Translating these into binary 0 + 1440 equals 0010110100000 and 0 + 1500 equals 0010111011100. Corresponding outputs from the frequency dividers uniquely define the time for initiation and termination of the subject operation as shown in figure 17. The signal derived from the second flip-flop is used to provide a quarter second delay and thus avoid problems associated with finite pulse propagation time. Certain simplifications are possible in this arrangement such as the omission of final zero inputs in the set function. This fortuitous coincidence occurs because these inputs can only change to l's with time and the output flip-flop remains in the set state during this time. This is also true of the final zeros in the reset function. These simplifications result in a small saving of hardware and therefore weight. It is possible to effect such savings in cases when they would not normally occur by trading time accuracy - the accuracy with which time of occurence approximates design center - for hardware saving.

It is to be pointed out that the system described here is extremely light in weight, very compact, very reliable, and capable of one-eighth second accuracy. Programmer No. I is similarly light, reliable and compact. Both are completely solid-state devices with the possible exception of the thermostat. The thermostat used in the experimental model contained moving contacts. Although it has not been tried

experimentally as part of this project, it is felt that a thermistor will function as well or better and perform its role more reliably. Moreover, its continuous control will permit external adjustment of both the end point and hysteresis of the thermostat. The clock of Programmer No. II will provide outputs of time in precisely the proper form for coding the instant of occurence of gas component peaks if this should be desirable.

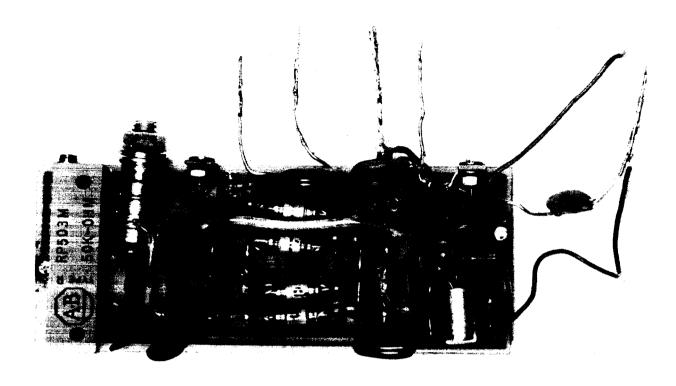
A possible alternate timing programmer could consist simply of a ramp generator with Schmidt triggers to sense the instant of intersection of the ramp with a preset voltage. The Schmidt triggers could control driver amplifiers or flipflops to produce the output commands. This scheme is more attractive than the digital clock, from the stand point of simplicity. The ramp generator could replace several flipflops thereby reducing the number of gate inputs required. For the saving in coincidence gate diodes to overcome the additional weight of the trigger circuits, the rate of change of ramp voltage would have to be so slow that the uncertainty of intersection time would probably exceed the quantizing steps of the digital clock. Both schemes, however, deserve investigation and should be considered further in the next phase of the effort.

3.4.4 <u>Electrometer Amplifier</u>

The electrometer amplifier as it now exists incorporates

as its control component a solid state operational amplifier. This design philosophy; should be adhered to for future considerations, since it appears to represent the best technique for maintaining the highest possible reliability in the face of the rather stringent environmental conditions. Hence, concerning the operational amplifier design in particular, it is recommended that it only be modified to the extent of providing a unit of comparable performance, but that it incorporate reliable electronic components, such as those listed in the JPL preferred parts list. The present amplifier incorporates a carrier frequency of 5 megacycles and some effort is expected to be required to assure that this does not produce any significant r-f interference with other system components. By proper shielding and capacity bypassing this requirement should be readily achieved. Circuit boards for the P-2 operational amplifier shown in figure 18 illustrate the light weight compact nature of the present solid-state package.

Our laboratory experiments and theoretical considerations of the automatic zeroing achieved by incorporating a linear electrical feedback network to generate a transfer response that greatly suppresses low frequency fluctuations, while passing unattenuated the frequency components important to faithful reproduction of the chromatographic response, appears particularly promising. In selecting this method for electrometer



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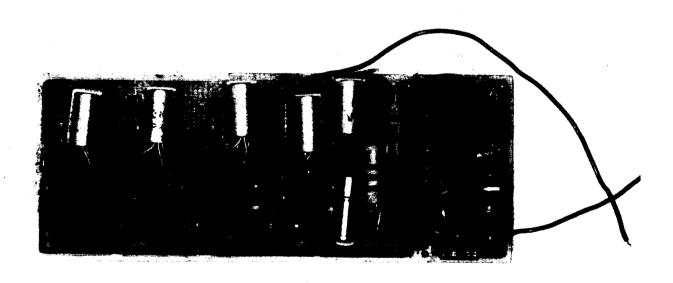


Figure 18. Photographs of Circuit Boards for P-2 Amplifier

zero correction, both electronic and electromechanical servo correction methods were considered and abandoned because of undue complexity and the ensuing possibility of power reliability. We believe that the method we have selected is the most suitable for the specific application, and that it achieves the desired goal simply and efficiently.

Finally, our present system incorporates two separate electrometer amplifiers to which 4 detector cells are connected in pairs. This arrangement is necessary to achieve distinct separation of various elution peaks. It is recommended that some further consideration be given to methods of reducing the requirement to only one electrometer: this might be done by further study of the column design and total analysis time to achieve sufficiently great separation of the elution peaks to permit their passage through a single common electrometer amplifier for a multiplicity of detectors. At this time the overlap of the skirts of the elution peaks with an analysis time frame of 5 minutes prevents the use of this concept; it can be also employed only at the expense of loss of accuracy of concentration or loss of a minor peak.

3.4.5 Logarithmic Amplifier

The principal function of the logarithmic amplifier is to compress the range of variation for the chromatographic response so that it may more readily be accommodated by the transmission link. However, because of at least two

considerations, other than simplification of the overall system, it may be advisable to eliminate the logarithmic amplifier from the design. First, the logarithmic characteristic of the present detectors already significantly compresses the range of variation of the signal output. Our present experience indicates that the current peaks range from 10^{-11} to 10^{-8} amps on a base line current of approximately 10^{-8} amp. Furthermore, our electrometer amplifier is noise limited by the random noise level of 10^{-12} amperes in the baseline current with very little or no contribution from the amplifier itself. Hence, an entire 60 db dynamic range is advisable to preserve the information in the peaks.

The next important consideration concerns the actual necessity of logarithmic compression for transmission. It is a fact that to optimumly feed the information to earth over a binary coded digital transmission link requires logarithmic conversion. Otherwise, transmission capacity will be disproportionate and hence wasted. However, rather than performing this conversion in the chromatograph sonde it would probably be more efficiently and accurately performed as part of the analog to digital conversion process in the parent vehicle with little extra complication in the A to D converter. Therefore, if the signal can be relayed to the A-D converter probably in the parent vehicle with good signal to noise ratio and dynamic range, i.e. 60 db, then there is probably no cause to

burden the chromatographic sonde electronic package with logarithmic converters. On the other hand if the dynamic range of the relay data link is not sufficient to accommodate a 60 db range, then it will be advisable to have the log converter in the chromatograph sonde.

If the logarithmic converters are ultimately required, then some effort will have to be exerted to assure that the non-linear feedback diodes used to achieve the logarithmic response are temperature stabilized. This, it is felt, can be achieved by permitting this portion of the package to be fully sinked to the stabilized oven package.

3.4.6 Squib Firing Circuit

To fire the squibs that operate the gas injection valve, a simple RC circuit was designed and built consisting of a 250 microfarad electrolytic capacitor charged through a 6800 ohm resistor from a 28 volt dc source. The squib ignites within one millisecond with any voltage over 18 volts applied. Its igniter resistance is about ten ohms. Discharging the 250 microfarad capacitor through the squib resistance, the voltage remains above 18 volts for about one millisecond. On the assumption that discharging the capacitor of the above described circuit through the squib igniter would fire the squibs with acceptable reliability, it can be used as a basis for design of an automated firing system. A silicon controlled switch can be connected between the squib and battery return. The

SCR can be easily and reliably triggered by Programmer No. II. The problem of turning it off remains. Generally the squib igniter burns open. Should this characteristic prove not to be consistent enough to be relied on for current interruption, an inductor could be used to change the polarity of voltage momentarily and permit the SCS to stop conducting. This is conventionally done by connecting it in the lead to the controlled switches. The inductor and capacitor series resonate for one-half cycle. When the current stops to reverse, the SCR interrupts and the capacitor is recharged.

In practice the 250 microfarad capacitor and limiting resistor absorbed 4.1 milliamperes at 28 volts at the start of the charging process. Total power per charge is $V^2C = 784 \times 2.5 \times 10^{-4} = 1.96 \times 10^{-1}$ Joules. For eight firings with shorted squibs this would amount to 177 Joules, a trivial total load. In practice, less than 30% of the charge of the capacitor was spent before the squib igniters opened. This would limit the requirement to only 0.67 Joules.

3.4.7 Power Supplies

The present electronic components operate at plus and minus ten volts. This supply voltage is directly realizable from a 28 volt battery using direct coupled regulators. The present regulators have proved satisfactory for laboratory use. However, more sophisticated and more complex regulators giving better control are current practice and can be substituted

without any complications. Should the battery be externally grounded, instead of floating, the problems produced could amount to, at most, a dc to dc converter, a device now well within the state-of-the-art. If high voltage gas detector cells working up to a thousand volts applied EMF are considered in future designs, the high voltages similarly can be generated in a dc to dc converter.

3.5 Processing of Chromatograph Data for Transmission

3.5.1 General

It should be recognized that, at distances as great as that of the planet Mars from Earth, information communication should be performed in the most efficient means possible. This is necessary if the message relaying the scientific data collected by the Mars vehicle sensors is to be retrievable by receivers on Earth, from the thermal and atmospheric noise background, at a minimum of expense in terms of size and weight of the Mars vehicle telemetry transmitter.

Maximumly efficient utilization of the transmission information space is achieved by devoting the information space available to the encoding of the significant features of the event to be relayed, and wasting as little as possible of the information space on features that are either highly improbable or of trivial importance. For transmission of the chromatograph output, Melpar has been informed that 800 bits of information space is available and that this can be transmitted at a rate of approximately 1 bit per second. In the following discussion, we shall concern ourselves with the manner in which the chromatograph information should be encoded to employ the available information space for the most efficient transfer of data to Earth. The methods outlined for transmission of the chromatograph data are those recommended by Melpar, in the light of information made available by JPL,

concerning the general nature of the communication link.

At one time during the effort, it had been expected that due to non-monotonic detector cell characteristics, delete certain concentration levels for some gaseous samples would produce a double peaked characteristic in which the height of one peak and the depth of the trough between them could be significant characteristics for classification of the event. If this were the case, it would be necessary to transmit, in addition to the amplitude and time of occurrence of the initial maximum, the amplitude of the minimum that separates the peaks. Such a second class of events would hence require additional information per chromatographic response, at the expense of other compound determinations.

It is rather obvious that the occurrence of such double peaks should be avoided if maximum utilization of the information capacity of the communication link for transfer of the chromatographic response data to earth is to be achieved.

Hence, in Melpar's performance on the subject contract every effort was exerted to avoid the condition, and indeed it now appears that the condition can be avoided and such double peaks will not occur during the analysis. Therefore, our approach is now predicated on the occurrence of only single maximum responses.

In the following paragraphs, the chromatographic responses are examined for their significant information content, a

functional description of a data processor designed to extract significant information from the chromatographic response is presented, and the means of encoding this information in binary coded form are discussed.

3.5.2 <u>Significant Information Content of the Chromatographic Response</u>

The chromatograph response consists, in general, of a series of elution peaks occurring sequentially in time. The significant information contained in these peaks lies in knowledge of the amplitude of the maximum and its time of occurrence. The information capacity required for transmission of the data depends on the accuracy with which the data is to be preserved and the dynamic range that the parameter to be observed covers. For transmission of the maximum amplitude and time of occurrence it is advised that logarithmic encoding be employed. In this method of encoding the levels of quantizing are such that each step is a constant percentage of the magnitude of the observation made. Hence, all observations have the same percentage accuracy. For an n bit binary word, the quantizing accuracy with which a parameter ranging between the limits $A_0 < < A_1$ is

% Quantizing Accuracy =
$$\frac{1}{2} \left[\left(\frac{A_1}{A_0} \right)^{\frac{1}{2^n}} - 1 \right] \times 100$$

(where the error is considered to be one-half the value of a

quantizing interval.) The accuracy resulting for a number of dynamic range conditions and binary quantizing levels is illustrated in the following tabulation.

QUANTIZING ACCURACY AS A FUNCTION OF DYNAMIC RANGE AND BINARY CODE DIGITS

Dynamic Range	Number of Binary Digits			
	7 bits	8 bits	9 bits	
60 d b	2.7%	1.35%	0.67%	
40 db	1.8%	0.9%	0.45%	
20 db	0.9%	0.45%	0.23%	

Reference to the table illustrates the manner in which information capacity varies as a function of dynamic range and accuracy. From this table a variety of trade-offs are seen to be possible. It is our opinion that each amplitude maximum sample should be encoded with 8 bits over a 60 db dynamic range thus providing a mean error of 1.35%. If one wishes greater accuracy without increased channel capacity, it could be achieved at the sacrifice of dynamic range. For example the same 8 bits applied to a 40 db dynamic range results in a mean error of 0.9%. As yet another trade-off example, if one wishes to reduce the information rate, it may be accomplished either at the expense of quantizing accuracy or dynamic range. This is apparent in the tabulation, where for a 7 bit word one has among possible choices a 60 db dynamic range with 2.7% mean

error or a 40 db dynamic range with 1.8% mean error.

A similar situation exists for time of occurrence. Our results indicate that the peaks will occur in the range 10 < t ₹300 se∞nds. This is a 30:1 dynamic range and it can be quantized with a mean error of 1.35% with a 7 bit word or a mean error of 0.67% for an 8 bit word. For this, our present opinion is that 7 bit quantization per sample is required.

From the above discussion, it appears that 8 bits for amplitude and 7 bits for time information are necessary to retrieve the significant information from a single peak in the chromatograph response function. Hence 15 bits per sample appears to be the goal that should be sought. Reference to figures 5 and 6 of Volume II of this report reveals that 10 peaks (including air peaks) from each of the two detector channels are possible, hence giving a total of 20 possible peaks per sample run. If each of these is to be quantized with 15 bits, then each run will require a total capacity of 300 bits. If 800 bits is the maximum allocation, then it is seen that one can expect to return to earth the results of two complete analyses. It should be noted that by increasing the capacity allocation to 900 bits, if it should become possible, it is possible to return to earth the results of three analyses.

Let us now consider what would happen if one is willing to sacrifice accuracy for the sake of multiple analyses within the confines of 800 bits. If three analyses are to be performed, each with a complement of 20 compounds, then the number of bits per sample cannot be greater than 13. One might allow 7 bits for amplitudes and 6 bits for time. For a dynamic range of amplitudes of 60 db, the mean quantizing error for peak amplitudes would be 2.7%. If the dynamic range were reduced to 40 db, this mean quantizing error would reduce to 1.8%. The time base accuracy for the interval 10<t<300 seconds would be 2.7%. This trade-off does not appear particularly severe and should be seriously considered.

Let us now go one step further and consider the possibility of 4 complete analysis runs. If each is to allow for 20 possible peaks, then for each, 10 bits of information space is allowable within the overall limit of 800 bits. A possible split is 5 bits for amplitude giving a mean quantizing amplitude accuracy of 12% for 60 db dynamic range or 7.5% for 40 db dynamic range, and 5.5% mean accuracy for time base quantization in the range of 10 < t < 300 seconds. These results appear to have excessive error.

Finally, if one could expect that out of the 20 possibilities only 15 will occur, then for four analyses, each sample could have 13 bits allocated to it and the situation previously described with 7 bits allocated to amplitude and 6 bits to time would prevail. This situation provided an amplitude quantizing accuracy of 2.7% for a 60 db dynamic range and a time accuracy

of 2.7% for the range from 10 to 300 seconds. This possibility appears quite feasible.

3.5.3 Functional Description of Data Processor

In this section, we present a discussion of a relatively simple data processor that automatically samples the amplitude of a chromatograph peak and its time of occurrence. The principal unit of this data processor is the sampling pulse generator. This unit, which is shown in functional block diagram form in figure 19 determines the time of peak occurrence by first differentiating the incoming chromatograph analysis function and examining this derivative for negative going axis crossings. As shown in figure 19, these negative going axis crossings signal the attainment of the peak value by the input chromatographic function. The pulses produced by the circuit at this instant are employed to initiate the amplitude and time base samplings which characterize the significant information contained in the analysis.

The complete data processor proposed for the present two channel analysis system is shown in figure 20. It contains two separate peak sample pulse generators of the type described in the previous paragraph, one for operation on each of the electrometer channels designated as Channel A and Channel B for this discussion. The system permits the sampling of a maximum occurring in either channel by the same Analog Sampler and also indicates whether the sampling occurred in Channel A or Channel B.

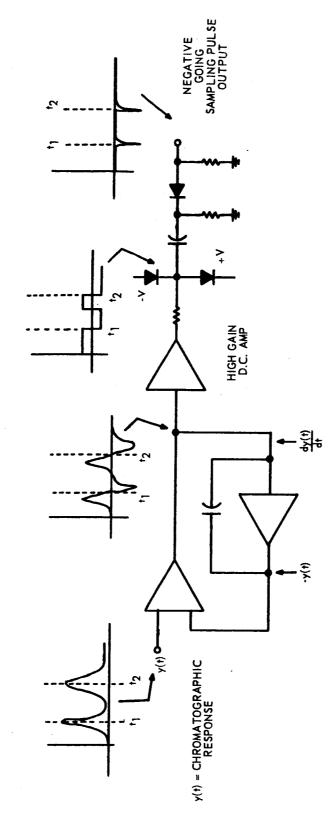


Figure 19. Block Diagram of Sampling Pulse Generator

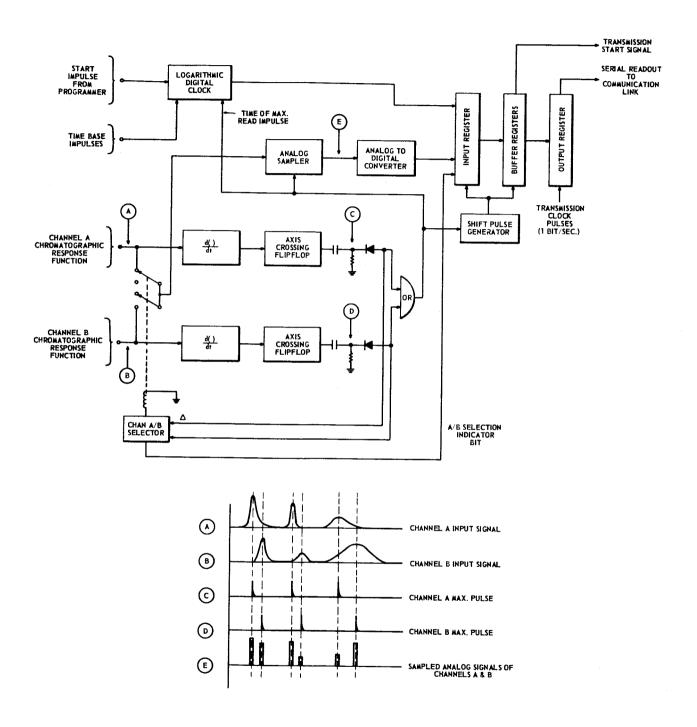


Figure 20. Dual Channel Chromatographic Data Processor

It is felt that the simultaneous occurrence of peaks in both channels A and B is very unlikely within the time resolution of the peak detector and sampling circuits. This time resolution can be made to be a very small fraction of a second and this is relatively insignificant compared to a response duration which is in terms of seconds.

Assuming now that the sampling epochs are properly generated, each peak will have its amplitude sampled by the analog sampler and this sample converted to binary code by the analog to digital converter. Also the time of occurrence of the peak will be established by sampling the time elapsed as indicated by the output of the digital clock. These binary coded samples of peak amplitude and time of occurrence along with the indication of the channel in which the peak occurred are fed to an input register and immediately transferred to a position in the buffer storage register. The buffer storage register could have sufficient capacity to store all chromatograph samples and later transmit them to the communication link upon command in serial form for transfer to the earth receiver. It should be noted that the channel classification could add an extra bit to each sample, and some means of avoiding this should be considered for maximum utilization of channel capacity. Information by position in the transmission frame could possible be employed, all responses coming from channel A be transmitted first, followed by those from channel B. Furthermore, some means of segmenting various analysis runs for multiple analyses must be considered. 83

4. INTEGRAL UNIT DESIGN

This section of the report deals with the integral design for all components of the unit. In particular, the problems of interconnection, space allocation, structural integrity, environmental survival and environmental tests are considered.

4.1 Weight and Volume of Sub Units

Table 5 provides a comprehensive summary of the projected individual volume and weights of the various components and subassemblies of the gas chromatograph. A fairly high level of confidence is reflected in the figures presented in view of the fact that many of the components are based on those developed for the laboratory The size and weight of the oven is based on figures for an oven of nearly the same size which was constructed and tested during the research phase. A discussion of the satisfactory performance of this oven was presented in another section of the report. The injector valve is a further modification of the injector valve which was constructed and used with satisfactory results in the laboratory model. The detectors were used in the laboratory model in a slightly larger configuration. The pressure regulator represents a slightly modified version of a similar pressure regulator, supplied by the Leonard Company, which has previously been used in other aerospace projects. Although the carrier gas reservoir has not been fabricated for the laboratory model, the figures presented are based on calculations related to the required size and wall thickness for the given pressure and temperature extremes. These calculations are derived from information given by manufacturers of titanium pressure vessels.

TABLE 5
WEIGHT AND VOLUME SUMMARY

SUBUNIT	VOLUME CU.IN.	WEIGHT LBS.
2 Logarithmic Amplifiers	2.0	0.10
2 Feed Back Amplifiers	2.0	0.10
2 Electrometer Amplifiers	4.0	0.20
2 Detector Power Supplies*	2.0	0.15
Feedback Control Circuit	5.0	0.30
Amplifier Power Supply	3.0	0.16
2 Programmers	15.0	0.50
Squib Control Circuit	3.0	0.12
Pressure Seals	0.45	0.10
2 Transit Seals	0.44	0.10
Pump (Venturi) (Al)	0.16	0.01
Carrier Gas Tank (Ti)	7.2	0.12
Regulator (Pressure)	3.5	0.27
Injector Valve	6.4	0.35
Oven (With Insul-Thermite etc.)	81.8	1.0
Sample Loop	1.0	0.06
Columns	2.45	0.11
4 Detectors	0.17	0.05
Case	7.56	1.12
Reference Gas Release	0.10	0.05

TABLE 5 (contd)

SUBUNIT	VOLUME CU.IN.	WEIGHT LBS.
Connecting Tubing	0.07	0.02
Wire (Cable)	0.17	0.03
	147.47	5.02

With a stacking factor of 1.545, Total 1.545 x 146.57 = 226 in³ Size 5.5 x 5.5 x 8.0 = 242 in³

^{*} There is a good probability that these power supplies will not be required. The 10 volt supply envisioned for the detectors may be taken from the amplifier power supplies.

The inlet exhaust ports and squib-actuated seals data are based on experimental laboratory models which were fabricated and tested during the research phase. The same applies to the critical orifice Venturi pump. The figures for the electronic circuitry including the amplifiers, programmers, and power regulators are derived from work performed during the research phase and from the extensive past experience by Melpar in packaging of miniaturized electronic circuitry for missiles for the defense industry. The assembled unit will assume a rectangular configuration measuring 5½ inches square by 8 inches long and weighing approximately 5 pounds. These figures indicate compliance with the specifications in regard to size and weight. However, further reduction of size and weight may very well be feasible during the development stage.

4.2 <u>Interconnection of Subunits</u>

The arrangement diagram given in figure 21 for the gas chromatograph reveals a general division of the components which: (1) must be temperature-controlled within close limits, (2) components which must be brought up to a certain minimum temperature, and (3) components which will not require preheating. Those components which are enclosed within the thermite oven and will require close temperature control are the chromatrograph columns, the detectors, and the injector valve. These components are arranged in a

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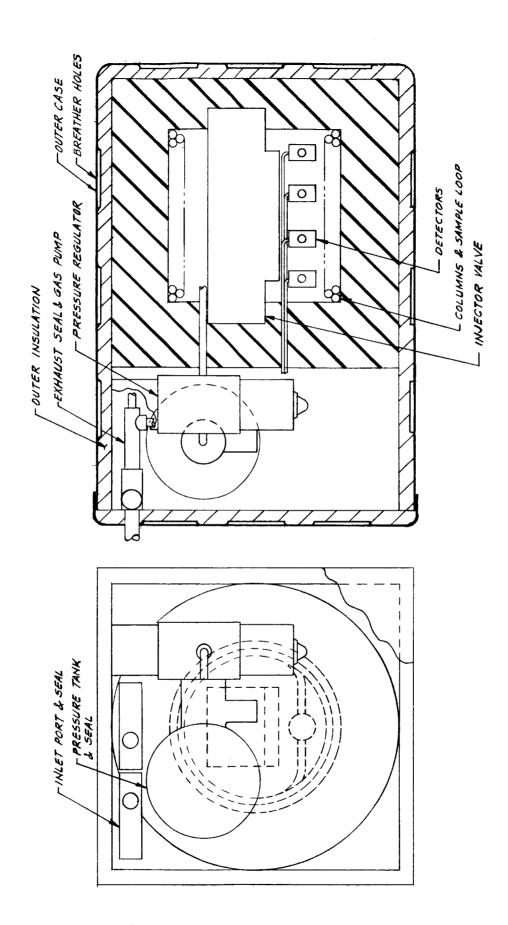


Figure 21a. Gas Chromatographic Breadboard Unit



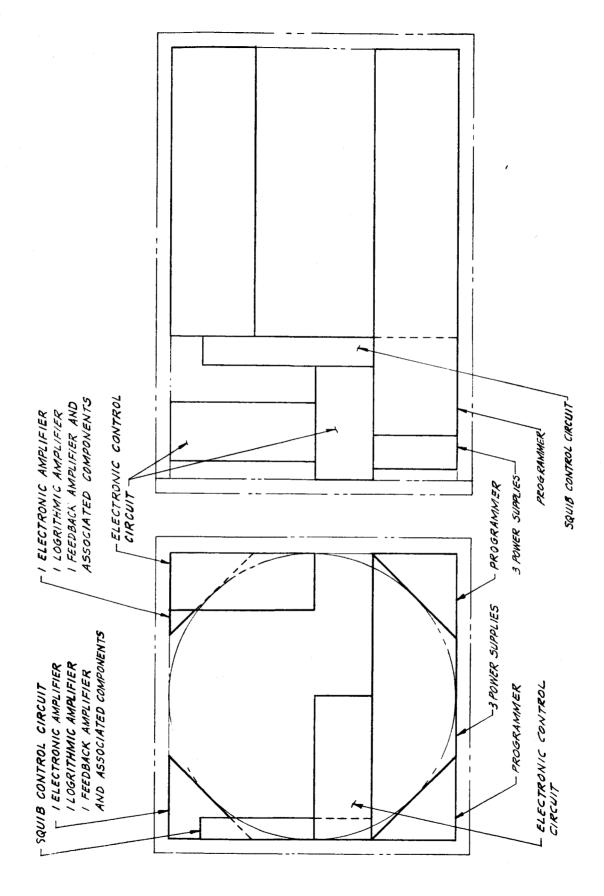


Figure 21b. Gas Chromatographic Breadboard Unit

cylindrical core surrounded by chemical units which, in turn, are surrounded by insulation material. The components which will require preheating to a minimum temperature prior to operation are the electronic and feedback amplifiers and the programmers. Logarithmic amplifiers, if used, will also require preheating. These electronic subassemblies will be enclosed in envelopes with a triangular cross section to fit into the corners of the space between the cylindrical oven and the rectangular outer shell. Electronic control circuitry, squib actuation circuitry, the pressure regulator carrier gas pressure tank, intake and exhaust ports, and gas pump will be in a compartment at the end of the rectangular enclosure at the end of the oven. These components will not require preheating prior to operation.

Interconnection of the chromatograph components within the oven is apparent from the flow control system diagram shown in figure 21. Principal emphasis here will be placed on the stages of assembly and proper interconnection in assuring that joints made in the flow system are hermetically sealed and do not protrude excessively. This section will be a preassembled as a subassembly and will be inserted into the oven with the thermites and insulation; interconnections will be used to the external components. The carrier gas pressure tank will be preassembled with the pressure regulator and mounted on a bulkhead divider plate over the oven.

Interconnections through the insulation to the internal components will be made by hermetically sealed joints which will be designed for minimum heat conductivity. The inlet port squib seal assembly and the exhaust port gas pump squib seal assembly will be mounted and interconnected to the internal oven section in a similar manner. A cable containing the leads from the detectors will lead through the insulation to the bulkhead section to make proper interconnections to the electronic circuitry. Electronic control circuits and squib circuits will be mounted to the top plate; the triangular sections containing the programmers and amplifiers and associated components will be fixed in the corners of the assembly. The integral unit will be enclosed with an outer case. For purposes of maintaining minimum weight and compaction, it is proposed that external electrical interconnection be accomplished by flying leads rather than by electrical connectors.

4.3 Structural Integrity

The value of an instrument for this type of application is strongly dependent on its ability to maintain structural integrity at blastoff, in transit, at Martian atmosphere injection, and at impact on Mars. The structural integrity must be such that its ultimate function as a Martian atmosphere analysis device is not impaired. The instrument proposed here encompasses means for eliminating all unnecessary

components in subassemblies and for achieving the ultimate in system performance with the highest reliability. The injection valve activated by squib firing is an example of a self-sufficient unit that does not require a bulky control valve such as a solenoid valve.

The components have been arranged in the breadboard configuration in a manner to provide the optimum protection of sensitive elements from any severe effects of the environ-The most critical components placed in the oven for temperature control are protected from shock and vibration to some extent by the insulation material used to reduce heat loss. In the assembly of the unit, direct coupling of the various subunits to one another will be carried out in such a way as to prevent cascading of assembly. The entire unit, when assembled, will be enclosed in a case made of a thin serrated metal which will contain a number of holes of microminiaturizing weight. The serrations will provide the rigidity necessary for the unit. Rapid ambient air pressure changes which may be experienced after launch and during injection at Mars could produce high stress on a case which does not provide for rapid breathing. Such breathing would be afforded by the holes.

4.4 Environmental Survival

The ultimate proof of the ability of the instrument to withstand the environments of extended space transit, exposure to zero gravity, temperature extremes, shock, and injection in the Martian atmosphere can only result from an extensive testing program. However, any of the problems may be anticipated on the basis of past experience with similar equipment used in missiles and satellites. Some of the more obvious problems which can be anticipated are the elimination of any functions which would depend on gravity, the elimination of as many as possible of the spring-actuated devices, and the elimination of any components which may be subject to internal pressures due to the vacuum conditions of space. Perhaps the most severe environmental restraint will arise from the extremes of temperature to which the instrument will be subjected. The possible low temperature of 140°K and the sterilization temperature of 145°C represent a very wide temperature range through which materials must operate properly. It is proposed, therefore, that, during the development of the breadboard mode, considerable effort be devoted to environmental design test work in the area of selection of materials for the system. This approach will aid in preempting any major road blocks in the transition from a breadboard model to a final prototype at a later date.

4.5 Environmental Tests

The JPL specification for the Mariner B probe and the gas chromatrgraph have been scrutinized to determine the feasibility of performing these tests at Melpar or the advisability of having some of the tests performed by regular subcontractors of Melpar. In Table 6 are paragraph numbers from the JPL specifications which are applicable to this work. The appropriate group that would handle the testing in indicated.

TABLE 6

CAPABILITIES REGARDING EXPERIMENTAL TESTING (Ref. JPL Spec. 30257 -- by paragraphs)

- 4.1.1.1 Temperature Test -- Thermal Sterilization can be accomplished at Melpar
- 4.1.1.2 Ethylene Oxide Gas Sterilization Test can be done at Melpar (if required)
- 4.1.1.3 Liquid Sterilants for Joints and Mating Surfaces may be done at Melpar if JPL requires (JPL Spec. questions the requirement)
- 4.1.2 Handling Shock
- 4.1.2a Drop Test

Can be done at Melpar

- 4.1.2b Bench Handling
- 4.1.3 Transportation Vibration

The 18 min. at 1.3 g from 2 to 10 cps would be subcontracted to:

Wyle-Parameters, Inc.,

Garden City Park, N.Y.

if true sinusoidal wave shape is required. If an approximate wave shape is satisfactory, it can be done at Melpar by use of a cantilever bar to achieve the required displacement. The balance of the test can be done at Melpar.

4.2.1 Explosive Atmosphere Environment -- Would be done at General Testing Laboratories at Moon

Archive, N.J.

4.2.2.1	Humidity
4.2.2.2	Oven Voltage Can be done at Melpar
4.2.2.2.1	Alternate
4.2.3	R-F Interference
	This work would be done by Melpar.
4.3.1	Static Acceleration
	Can be done at Melpar
4.3.2.la	Vibration - Low Frequency
	Melpar would improvise or subcontract to
	Wyle-Parameters
4.3.2.1b	High Frequency
	Can be done at Melpar
4.3.2.1c	Mid-Course Motor Vibration Test
	Not required for our unit but could be done
	at Melpar
4.4.la	Preferred Shock Test (see 4.4.1b)
4.4.1b	Acceptable Alternate
	At present, Melpar would have to subcontract
	to Aero Research and Development Corporation,
	Wilmington, Massachusetts. However, Melpar
	anticipates the purchase of suitable equipment
	to accomplish this test after the first of the
	year.
4.4.2	Space Flight Temperature

Can be simulated with a bell jar vacuum system from Melpar Research Laboratories. Melpar Environmental Laboratories are anticipating purchase of more elaborate equipment after the first of the year. Tests may be subcontracted to: Mount Vernon Research, Alexandria, Virginia Ref. JPL SW 2727

Part II

B2 Environmental Constraints

a - Sterilization - Melpar

cl - Vibration - Melpar

c2 - Shock - subcontract Aero Research and

Development Corporation

d - Temperature - Melpar

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met

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